employed as fillers in gelatin capsules; preferred materials in this connection also include lactose or milk sugar, as well as high molecular weight polyethylene glycols. When aqueous suspensions and/or elixirs are desired for oral administration the active ingredient may be combined with various sweetening or flavoring agents, coloring matter and, if so desired, emulsifying and/or suspending agents, together with such diluents as water, ethanol, propylene glycol, glycerin and various combinations thereof.

For parenteral administration, a solution of an active salt 10 in either sesame or peanut oil or in aqueous propylene glycol can be employed. The aqueous solutions should be suitably buffered (preferably pH greater than 8), if necessary, and the liquid diluent first rendered isotonic. These aqueous solutions are suitable for intravenous injection purposes. The 15 oily solutions are suitable for intraarticular, intramuscular and subcutaneous injection purposes. The preparation of all these solutions under sterile conditions is readily accomplished by standard pharmaceutical techniques well known to those skilled in the art.

It is also possible to administer the active salts topically and this can be done by way of creams, a patch, jellies, gels, pastes, ointments and the like, in accordance with standard pharmaceutical practice.

# **EXAMPLES**

The following examples illustrate the methods and compounds of the present invention. It will be understood, however, that the invention is not limited to the specific Examples.

## Example 1

L-Tartrate Salt of 5,8,14-Triazatetracyclo [10.3.1.0<sup>2,11</sup>.0<sup>4,9</sup>]-hexadeca-2(11),3,5,7,9-pentaene (Anhydrous Polymorph, Form B)

A speck-free vessel was charged with L-tartaric acid (780 55 grams, 1.1 equiv.) and methanol (7.5 L). The contents of the vessel were stirred until solution and speck free filtered into the crystallization vessel. 5,8,14-triazatetracyclo [10.3.1.0<sup>2,11</sup>.0<sup>4,9</sup>]-hexadeca-2(11),3,5,7,9-pentaene free base (992 grams) and methanol (7.5 L) were dissolved in the 60 vessel; the mixture was maintained at between 20 to 25° C. The solution of 5,8,14-triazatetracyclo[10.3.1.0<sup>2,11</sup>.0<sup>4,9</sup>]-hexadeca-2(11),3,5,7,9-pentaene free base was added over about 45 minutes to the L-tartaric acid solution through a filter to render the solution speck and fiber free. The product 65 was allowed to stir at 20 to 25° C. overnight and isolated by filtration. The product was dried under vacuum at 35 to 45°

C. to give 1618.4 grams (95.4%) of 5,8,14-triazatetracyclo [10.3.1.0<sup>2,11</sup>.0<sup>4,9</sup>]-hexadeca-2(11),3,5,7,9-pentaene L-tartrate salt Form B (MW 361.36). M.p. 210.5° C.; verified as Form B by powder x-ray diffraction.

#### Example 2

L-Tartrate Salt of 5,8,14-Triazatetracyclo [10.3.1.0<sup>2,11</sup>.0<sup>4,9</sup>]-hexadeca-2(11),3,5,7,9-pentaene (Anhydrous Polymorph, Form A)

A reactor was charged with 5,8,14-triazatetracyclo  $[10.3.1.0^{2,11}.0^{4,9}]$ -hexadeca-2(11),3,5,7,9-pentaene free base (2 g; 0.0095 mole, 1.0 equiv.) and methanol (60 mL, 30 mL/g). The mixture was stirred at 20 to 25° C. until completely dissolved. A second reactor containing a solution of L-tartaric acid (1.55 g, 0.0103 mole, 1.1 equiv.) dissolved in methanol (60 mL, 30 mL/g) was heated to reflux in methanol (i.e., 60 to 66° C.). The free base solution was added to the L-tartaric acid solution at methanolic reflux temperature over 20 minutes. The resulting slurry was cooled to 20 to 25° C. over a 1 hour period. The reaction mixture was allowed to stir for approximately 2 hours followed by isolation of the product by filtration. The solid product was washed with methanol (10 mL), then dried under vacuum at 30 to 35° C. to give 3.3 grams (97%) of 5,8,14-triazatetracyclo[10.3.1.0<sup>2,11</sup>.0<sup>4,9</sup>]-hexadeca-2(11),3, 5,7,9-pentaene L-tartrate Form A. The identity as Form A was determined by PXRD as compared with standard samples.

### Example 3

L-Tartrate Salt Form C of 5,8,14-Triazatetracyclo [10.3.1.0<sup>2,11</sup>.0<sup>4,9</sup>]-hexadeca-2(11),3,5,7,9-pentaene (Form C)

Preparation of CP-526,555-18 Form C from Form A or Form B:

L-tartrate salt Form B (~5 g) was dissolved in water (10 to 15 ml). Acetonitrile (200 to 300 ml) was added and Form C formed as a white precipitate. The resulting slurry was allowed to stir for 10 minutes and then filtered. The wet cake was then allowed to air dry. Product was determined to be Form C by NIR spectroscopy, DSC and PXRD analysis. This procedure may be run with Form A to yield Form C.

### Example 4

L-Tartrate Salt Form A of 5,8,14-Triazatetracyclo [10.3.1.0<sup>2,11</sup>.0<sup>4,9</sup>]-hexadeca-2(11),3,5,7,9-pentaene (Form A)

Preparation of Form A from Form C: L-tartrate salt Form C (~2 g) was added to 200 to 300 mL hot ethanol (~75° C.) and allowed to stir for 30 minutes. The sample was filtered hot and then dried in a 45° C. vacuum oven (house vacuum). The material was determined to be Form A by NIR spectroscopy, DSC, and PXRD analysis.

What is claimed is:

- 1. The tartrate salt of 5,8,14-triazatetracyclo [10.3.1.0<sup>2,11</sup>.0<sup>4,9</sup>]-hexadeca-2(11),3,5,7,9-pentaene.
- 2. A compound according to claim 1 which is the L-tartrate salt.
  - 3. A compound according to claim 2 which is anhydrous.
- 4. A compound according to claim 1 which is the D,L-tartrate salt.
- 5. A compound according to claim 4 which is anhydrous.
- 6. A compound according to claim 1 which is D-tartrate